PI 1121243

# THE UNITED STATES OF AMERICA

TO ALL TO WHOM THESE PRESENTS SHALL COME:

**UNITED STATES DEPARTMENT OF COMMERCE**
**United States Patent and Trademark Office**

**February 02, 2004**

**THIS IS TO CERTIFY THAT ANNEXED HERETO IS A TRUE COPY FROM THE RECORDS OF THE UNITED STATES PATENT AND TRADEMARK OFFICE OF THOSE PAPERS OF THE BELOW IDENTIFIED PATENT APPLICATION THAT MET THE REQUIREMENTS TO BE GRANTED A FILING DATE.**

**APPLICATION NUMBER:** *60/435,071*
**FILING DATE:** *December 20, 2002*
**RELATED PCT APPLICATION NUMBER:** *PCT/US03/38735*

**By Authority of the**
**COMMISSIONER OF PATENTS AND TRADEMARKS**

N. WOODSON
**Certifying Officer**

**PRIORITY DOCUMENT**
SUBMITTED OR TRANSMITTED IN COMPLIANCE WITH RULE 17.1(a) OR (b)

12-23-02 60435071.122002 Pro

PTO/SB/16 (10-01)
Approved for use through 10/31/2002. OMB 0651-0032
U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE

Under the Paperwork Reduction act of 1995, no persons are required to respond to a collection of information unless it displays a valid OMB control number.

# PROVISIONAL APPLICATION FOR PATENT COVER SHEET

This is a request for filing a PROVISIONAL APPLICATION FOR PATENT under 37 CFR 1.53(c)

| Express Mail Label No. | EL 933187342 US |
|---|---|

| INVENTOR(S) | | |
|---|---|---|
| Given Name (first and middle [if any]) | Family Name or Surname | Residence (City and either State or Foreign Country) |
| JAMES A. | FAUNCE | NORTH AURORA, IL 60542 |

☐ *Additional inventors are being named on the _____ separately numbered sheets attached hereto*

**TITLE OF THE INVENTION (500 characters max)**

HYDROLYTICALLY STABLE PHTHALATE ESTER LUBRICANTS AND METHOD OF METAL WORKING WITH HYDROLYTICALLY STABLE PHTHALATE ESTERS LUBRICANTS

*Direct all correspondence to:* **CORRESPONDENCE ADDRESS**

☒ Customer Number [ ] → *Type Customer Number here*

*OR*

23446
PATENT TRADEMARK OFFICE

| | | | | | |
|---|---|---|---|---|---|
| ☐ Firm or Individual Name | MCANDREWS, HELD & MALLOY, LTD. | | | | |
| Address | 500 WEST MADISON STREET | | | | |
| Address | 34TH FLOOR | | | | |
| City | CHICAGO | State | ILLINOIS | Zip | 60661 |
| Country | USA | Telephone | (312) 775-8000 | Fax | (312) 775-8100 |

**ENCLOSED APPLICATION PARTS *(check all that apply)***

☒ Specification *Number of Pages* 11
☒ Drawing(s) *Number of Sheets* 5
☐ Application Data Sheet. See 37 CFR 1.76
☐ CD(s) *Number* [ ]
☐ Other (specify) [ ]

METHOD OF PAYMENT OF FILING FEES FOR THIS PROVISIONAL APPLICATION FOR PATENT

☐ Applicant claims small entity status. See 37 CFR 1.27.
☒ A check or money order is enclosed to cover the filing fees
☐ The Commissioner is hereby authorized to charge filing fees or credit any overpayment to Deposit Account Number: 13-0017
☐ Payment by credit card. Form PTO-2038 is attached.

FILING FEE AMOUNT($): 160

The invention was made by an agency of the United States Government or under a contract with an agency of the United States Government.

☒ No.
☐ Yes, the name of the U.S. Government agency and the Government contract number are: _____

*Respectfully submitted,*

SIGNATURE [signature]
TYPED or PRINTED NAME ALEJANDRO MENCHACA
TELEPHONE (312) 775-8000

Date DECEMBER 20, 2002
REGISTRATION NO. *(if appropriate)* 34,389
Docket Number: 14154US01

## USE ONLY FOR FILING A PROVISIONAL APPLICATION FOR PATENT

This collection of information is required by 37 CFR 1.51. The information is used by the public to file (and by the PTO to process) a provisional application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 8 hours to complete, including gathering, preparing, and submitting the complete provisional application to the PTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, Washington, D.C. 20231. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Box Provisional Application, Assistant Commissioner for Patents, Washington, D.C. 20231.

[redacted]

"Express Mail" mailing label number: EL 933187342 US

Date of Deposit: DECEMBER 20, 2002

Attorney Docket No. 14154US01

# HYDROLYTICALLY STABLE PHTHALATE ESTER LUBRICANTS AND METHOD OF METALWORKING WITH HYDROLYTICALLY STABLE PHTHALATE ESTERS LUBRICANTS

## RELATED APPLICATIONS

**[0001]** [Not Applicable]

## FEDERALLY SPONSORED RESEARCH OR DEVELOPMENT

**[0002]** [Not Applicable]

## [MICROFICHE/COPYRIGHT REFERENCE]

**[0003]** [Not Applicable]

## BACKGROUND OF THE INVENTION

**[0004]** The metalworking industry requires lubricants in many of its operations. Water-based lubricants are particularly desirable because of the ease of using and disposing of the water base. Adipate esters have also been used as successful lubricants in the metalworking industry. Adipate esters, however, are unstable in water solutions and thus less desirable than water-based lubricants. In the past, it has been determined that esters having groups with steric bulk immediately adjacent the ester group may lend some hydrolytic stability to lubricants. The metalworking industry has gone to utilizing isopropyl and 2-ethylhexyl esters as a source of hydrolytic stability due to the steric bulk of the groups adjacent to the ester linkage. However, there continues to be a need for hydrolytically stable lubricants for use in the metalworking industry.

## BRIEF SUMMARY OF THE INVENTION

**[0005]** The present invention provides a hydrolytically stable phthalate ester lubricant and a method of metalworking with a hydrolytically stable phthalate ester lubricant. The phthalate esters used in the present invention have shown superior hydrolytic stability versus other esters and have shown successful lubrication results in ASTM testing for lubricants.

## BRIEF DESCRIPTION OF SEVERAL VIEWS OF THE DRAWINGS

**[0006]** Figure 1 compares the hydrolytic stability of the propoxylated Stepanpol PS-2002 with adipic ester in an acidic system.

**[0007]** Figure 2 demonstrates the hydrolytic stability of adipate polyol in a basic system.

**[0008]** Figure 3 demonstrates the hydrolytic stability of the propoxylated Stepanpol PS-2002 in a basic system.

**[0009]** Figure 4 illustrates the hydrolytic stability of adipate polyol in a KOH system.

**[0010]** Figure 5 illustrates the hydrolytic stability of the propoxylated Stepanpol PS-2002 in a KOH system.

## DETAILED DESCRIPTION OF THE INVENTION

**[0011]** The phthalate esters that may be used in the lubricant of the present invention include (I) a phthalate polyester-ether polyol, (II) a phthalic anhydride reacted with an equivalent of a fatty alcohol which is then ethoxylated with a variety of moles of ethylene oxide (or propylene oxide), and (III) the amine-neutralized salts of item (II).

**[0012]** Referring to (I), the phthalate polyester-ether polyol is the reaction product of:

(1) about 2 – 60 % based on the weight of the polyester-ether polyol of phthalic anhydride or phthalic acid;

(2) about 40 - 98 % based on the weight of the polyester-ether polyol of at least one polyol of the formula:

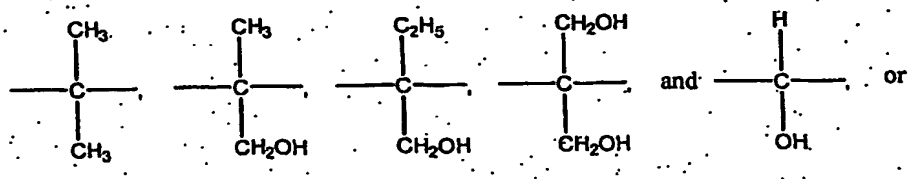
$$HO\text{-}R_1\text{-}OH$$

wherein $R_1$ represents:

(a) alkylene groups of about 2 to 10 carbon atoms;

(b) $-CH_2\text{-}R_2\text{-}CH_2-$

where $R_2$ represents:

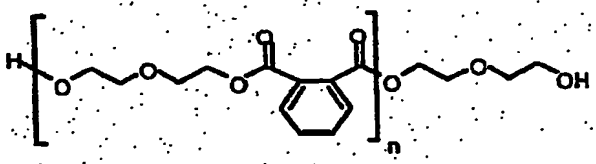


(c) $-(R_3O)_n-R_3-$

where each $R_3$ independently is an alkylene group of about 2 to about 4 carbon atoms, and n is an integer of from about 1 - 200; and

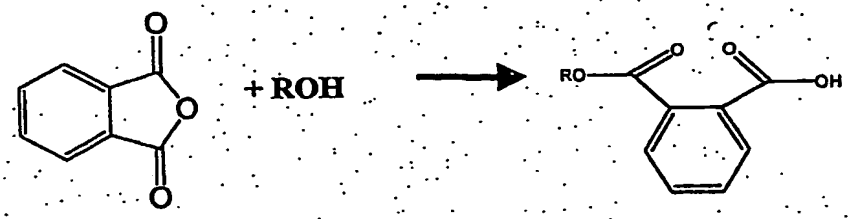
(3) about 10 – 80 % based on the weight of the polyester-ether polyol of an alkoxylating agent.

**[0013]** A preferred phthalate polyester-ether polyol is a propoxylated diethylene glycol-phthalic anhydride-based polyester polyol. The diethylene glycol-phthalic anhydride-based polyester polyol is sold by the Stepan Company under the tradename Stepanpol PS-2002. Stepanpol PS-2002 has the following structural formula.



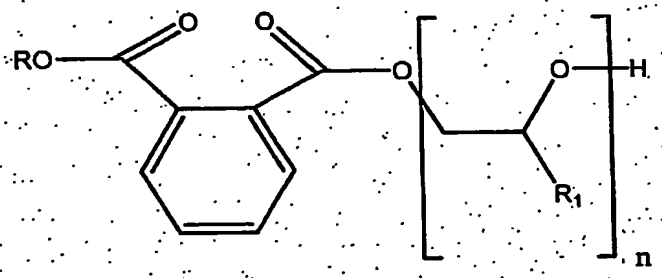
( n ~ 2.0)

[0014] Regarding (II), the phthalic anhydride - fatty alcohol reaction is described as follows.




[0015] where R is $C_4$ to $C_{22}$, branched or linear.

[0016] The ethoxylation reaction of the product of the phthalic anhydride - fatty acid reaction proceeds by ethoxylating the product with a variety of moles of ethylene oxide (or propylene oxide or butylene oxide) resulting in the following structure.




where $R_1$ = H, $CH_3$, $CH_2CH_3$, and n = 1-20, preferably 1-10.

[0017] Regarding (III), the amine-neutralized salts of (II) can also be used as the phthalate ester in the present invention. The amines that may be used to accomplish this neutralization include triethanolamine, triethylamine, triethanolamine, monoethanolamine, 2-ethylhexylamine, tallow amine ethoxylates or any other amine in general.

[0018] The stability of the phthalate esters of the present invention was investigated in both acidic and basic conditions and compared to adipic ester, a known lubricant often used in the metalworking industry.

[0019] Figure 1 compares the hydrolytic stability of the propoxylated Stepanpol PS-2002 with adipic ester in an acidic system. Figure 1 depicts the increase in acid value of the adipic ester versus the propoxylated Stepanpol PS-2002 as the acid value increase is indicative of ester breakdown. The results reported in Figure 1 confirm that the phthalate esters of the present invention provide the hydrolytic stability in acidic conditions desirable for lubricants in the metalworking industry.

[0020] Figure 2 and Figure 3 demonstrate the hydrolytic stability of adipate polyol and the propoxylated Stepanpol PS-2002, respectively, in a basic system. Base stability can be observed by measuring the breakdown in molecular weight of the ester over time. Figures 2 and 3 illustrate the breakdown in molecular weight via gel permeation chromotography (GPC) of adipate polyol and the propoxylated Stepanpol PS-2002, respectively, in 0.50M KOH and 0.50M TEA (triethyl amine) by measuring the area percent of the highest molecular weight species in the GPC graph. Figure 2 demonstrates that the area percent of the adipate polyol decreases drastically over time. In contrast, Figure 3 demonstrates that the area percent of the propoxylated Stepanpol PS-2002 does not decrease over the same time period.

[0021] Figure 4 and Figure 5 illustrate the hydrolytic stability of adipate polyol and the propoxylated Stepanpol PS-2002, respectively, in a KOH system. Figures 4 and 5 directly measure the average molecular weight of the systems to demonstrate the respective stability in a basic system. Figure 4 demonstrates that the average molecular weight of the adipate polyol decreases drastically over time. In contrast, Figure 5 demonstrates that the average molecular weight of the propoxylated Stepanpol PS-2002 does not decrease over the same time period, confirming the hydrolytic stability of the pthalate ester

[0022] All of Figures 2, 3, 4, and 5 confirm that the phthalate esters of the present invention provide the hydrolytic stability in basic conditions desirable for lubricants in the metalworking industry.

[0023] The lubricant of the present invention was compared to metalworking industry lubricant standards (isopropyl oleate and 2-ethylhexyl oleate). In particular, the tests run

were a 4-ball wear, Extreme Pressure Pin and Vee, and a Tapping Torque. All tests were run via an ASTM method.

**[0024]** Table I sets forth the results for the ASTM D-4172 4-ball wear testing.

TABLE I

| Product | Wear Diameter (mm) |
|---|---|
| Stepan MWA-560 HS | 0.598 |
| 2-ethylhexyl oleate | 0.752 |
| isopropyl oleate | 0.628 |

**[0025]** Table II sets forth the results for the ASTM D-3233 Extreme Pressure Pin and Vee testing.

TABLE II

| Product | Failure Load (lbs) |
|---|---|
| Stepan MWA-560 HS | 1000 |
| 2-ethylhexyl oleate | 1250 |
| isopropyl oleate | 1250 |

**[0026]** Table III sets forth the results for the ASTM D-5619 Tapping Torque testing.

TABLE III

| Product | Efficiency (%) |
|---|---|
| Stepan MWA-560 HS | 96.20 |
| isopropyl oleate | 110.28 |

[0027] The results as set forth in Tables I, II, and III demonstrate that the lubricants of the present invention provide lubricating properties comparable to those of the metalworking industry standards. As such, the lubricants of the present invention may be utilized in any application in the metalworking industry that requires a lubricant such as isopropyloleate or 2-ethylhexyl oleate.

[0028] A typical lubricant formulation useful in the metalworking industry may consist of up to about 90% water, about 5% phthalate ester, and about 5% triethanolamine. Those skilled in the art recognize that many lubricant formulations are maintained as proprietary trade secret information, and that the phthalate esters disclosed above may be utilized as the main lubricating ingredient in those proprietary lubricant formulations. Thus, the present invention includes such lubricant formulations that utilize the phthalate ester as the main lubricating ingredient. In other words, a person skilled in the art may take his or her proprietary formulation and in the place of the prior main lubricating ingredient utilize the phthalate esters to arrive with a lubricant formulation of the present invention. Such proprietary formulation usually include water, the main lubricating ingredient and at least one other ingredient. The at least one other ingredient may be a single ingredient, as described in the formulation above where the at least one other ingredient is triethanolamine, or may include any number of components.

[0029] Typical water-based lubricants presently include water in an amount between about 60% and about 93% by weight of the total composition, preferably about 75 to about 87%, and a main lubricating ingredient in an amount between about 2% and about 20% by weight of the total composition, preferably about 5 to about 10%. The phthalate esters disclosed above may be used as the main lubricating ingredient in such water-based lubricants and in such amounts. Typical water-based lubricants presently further include at least one other ingredient in a total weight of about 2% and about 20% based on the final composition. One typical component of the at least one other ingredients is an amine in an amount of between about 2% and about 10% by weight of the total composition. The amine is typically used to regulate the pH of the lubricant. Thus, the phthalate esters disclosed above can be used in

presently used lubricants by replacing the main lubricating ingredient in those present day lubricants with the disclosed phthalate esters to provide the lubricant of the present invention.

**[0030]** The phthalate esters disclosed above may also be used as the main lubricating ingredient in such water-based lubricants including this at least one other ingredient.

**[0031]** The invention has been described with reference to preferred and alternate embodiments. Modifications and alterations will occur to others upon the reading and understanding of the specification. It is intended to include all such modifications and alterations insofar as they come within the scope of the appended claims or equivalents thereof.

[0032] Claims

1. A method of making a water-based lubricant comprising the steps of

providing a phthalate ester;

providing water; and

mixing the phthalate ester and water.

2. The method of claim 1 further comprising the steps of

providing at least one other desirable ingredient; and

mixing the phthalate ester, water and at least one other desirable ingredient.

3. The method of claim 1 wherein the phthalate ester is provided in a amount of about 2% to about 20% based on the weight of the final composition; and

the water is provided in an amount of about 60% to about 93% based on the weight of the final composition.

4. The method of claim 2 wherein

the phthalate ester is provided in a amount of about 2% to about 20% based on the weight of the final composition;

the water is provided in an amount of about 60% to about 93% based on the weight of the final composition; and

the at least one other desirable ingredient is provided in a total amount of about 2% to about 20% based on the weight of the final composition.

5. A water-based lubricant comprising

phthalate ester in an amount of about 2% to about 20% based on the weight of the final composition;

water in an amount of about 60% to about 93% based on the weight of the final composition.

6. The water-based lubricant of claim 5 further comprising

at least one other desirable ingredient in a total amount of about 2% to about 20% based on the weight of the final composition.

7. The water-based lubricant of claim 6 wherein the phthalate ester comprises 5% by weight of the final composition; water comprises 90% by weight of the final composition; and the at least one other desirable ingredient comprises a total of 5% of the final composition.

8. The water-based lubricant of claim 5 wherein the at least one other desirable ingredient is triethanolamine.

9. A method of metalworking comprising the step of

utilizing a water-based lubricant comprising

phthalate ester in an amount of about 2% to about 20% based on the weight of the final composition;

water in an amount of about 60% to about 93% based on the weight of the final composition.

10. The method of claim 9 wherein the water-based lubricant further comprises at least one other desirable ingredient in a total amount of about 2% to about 20% based on the weight of the final composition.

## ABSTRACT OF THE DISCLOSURE

A water-based lubricant is provided that includes a phthalate ester in an amount of about 2% to about 20% based on the weight of the final composition and water in an amount of about 60% to about 93% based on the weight of the final composition. Also provided are a method of making the water-based lubricant and a method of metalworking using the water-based lubricant.

# Figure 1

*Stability: 2% in water @95°C pH 6*

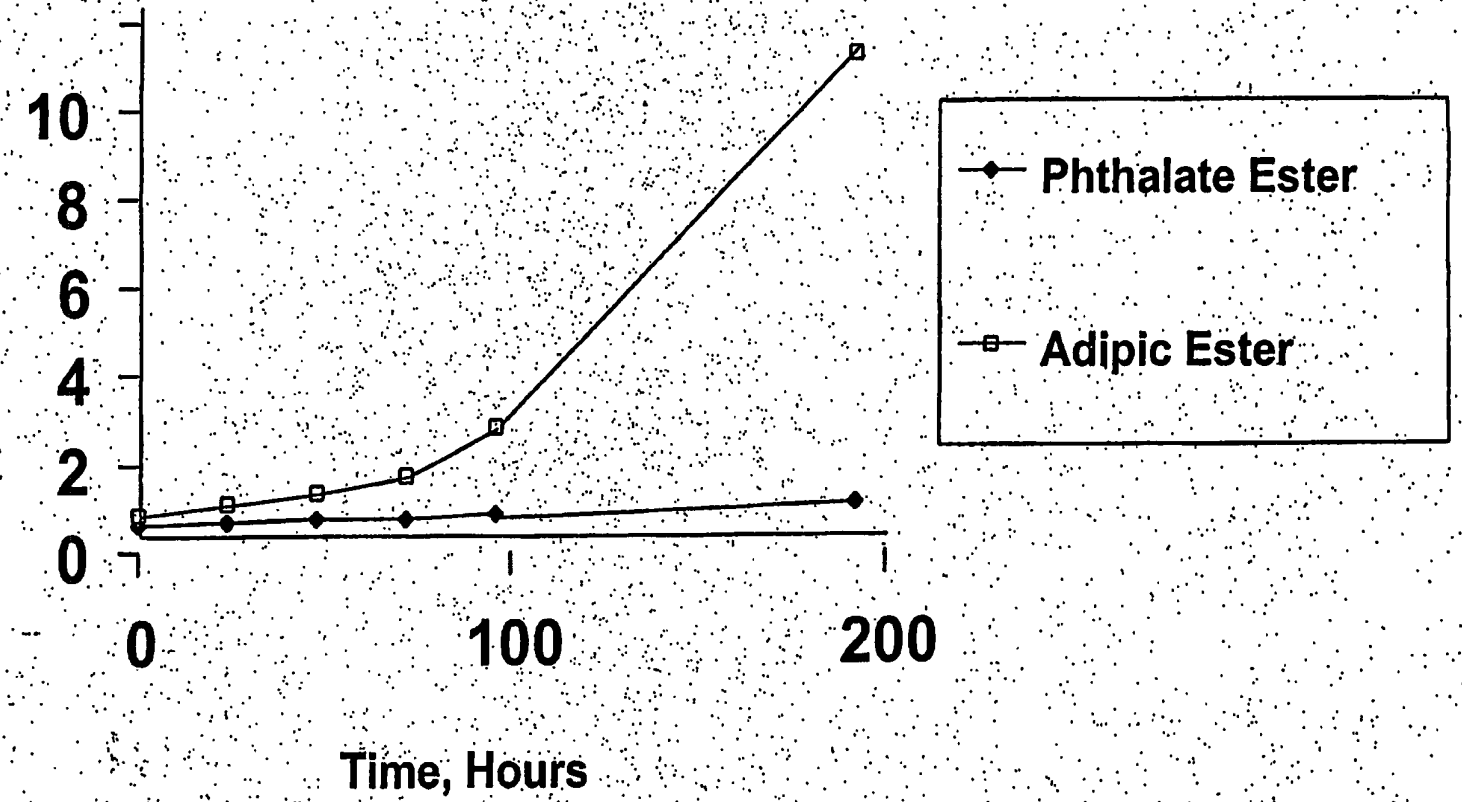
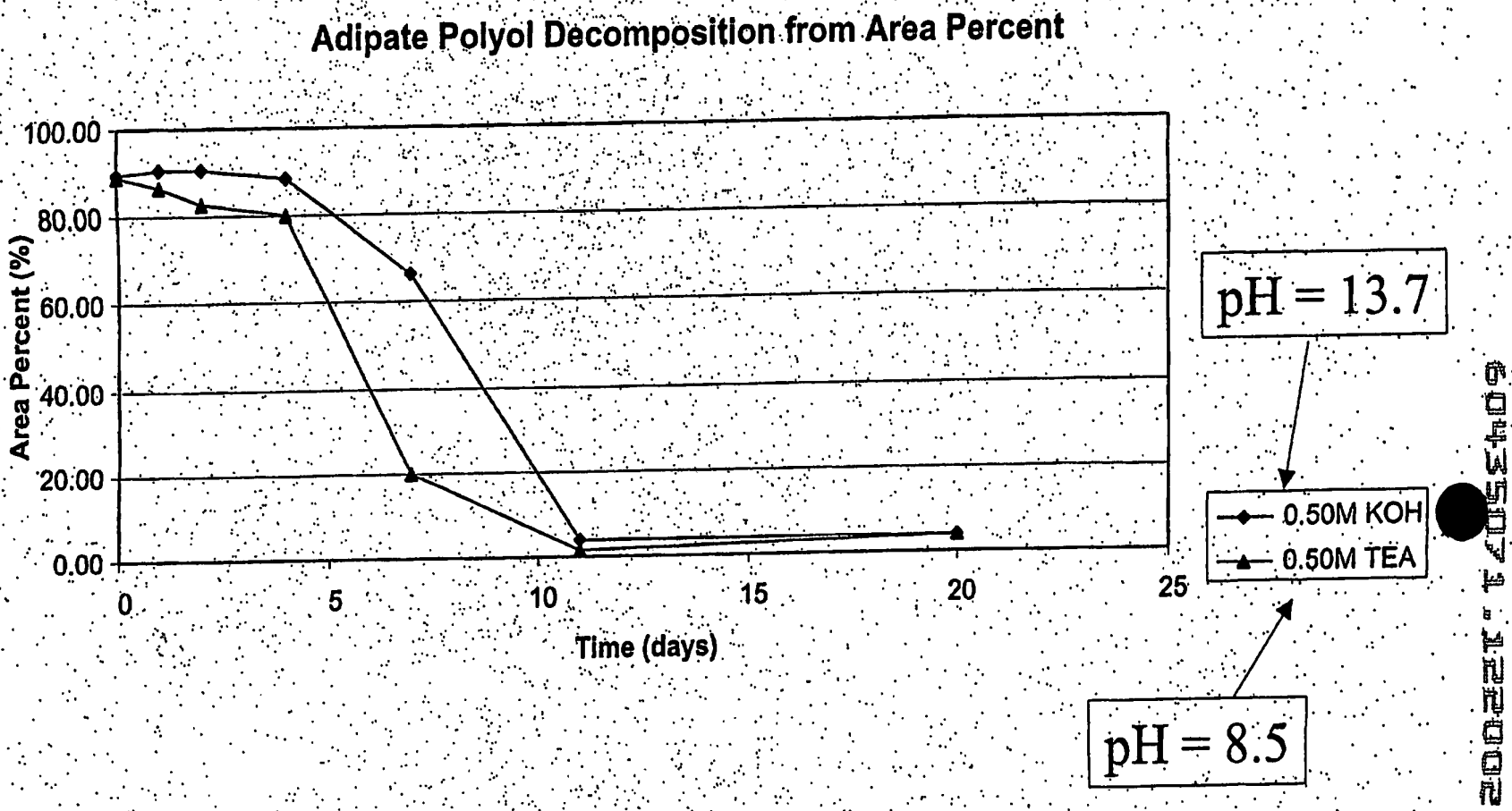


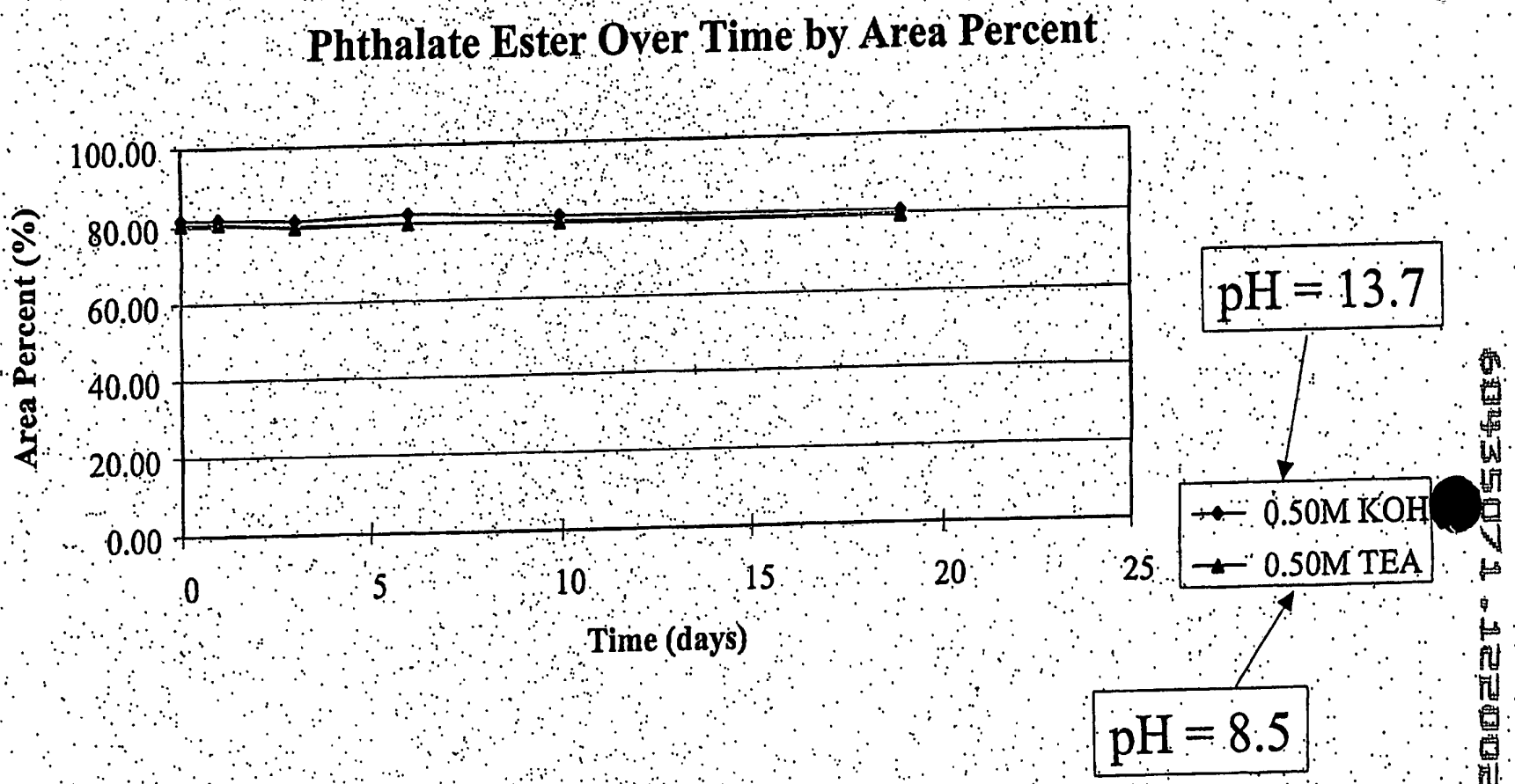
Figure 2

# *Stability: 70% in Base*



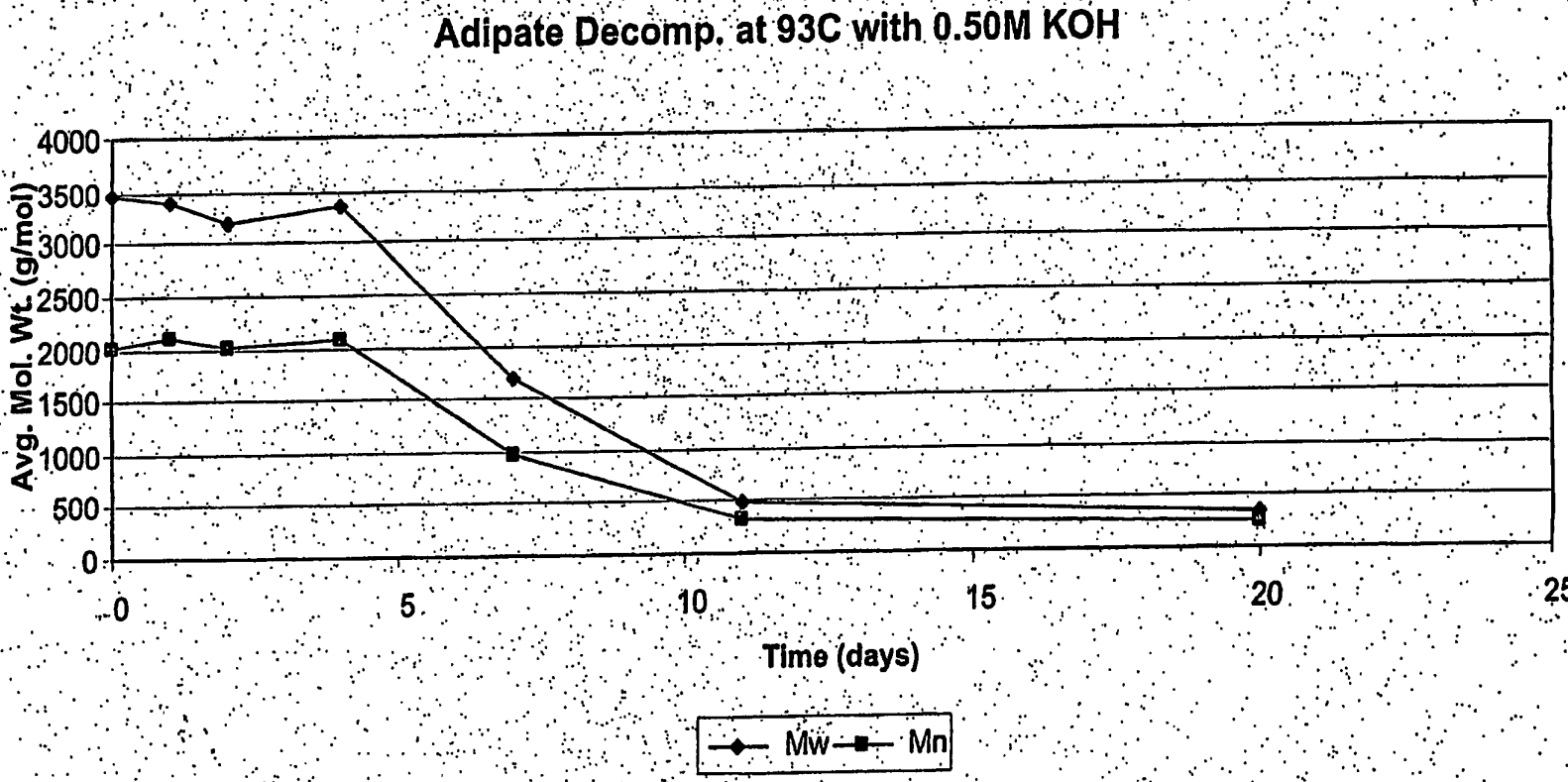
# Figure 3

## *Stability: 70% in Base*



# Figure 4

## *Stability: 70% in 0.5M KOH (13.7 pH)*



# Figure 5

## *Stability: 70% in 0.5M KOH (13.7 pH)*